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54 Title of the Invention Hair Cosmetic
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Specifications

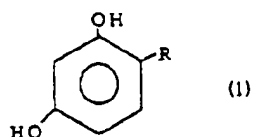
1. Title of the Invention

Hair Cosmetic

2. Claims

A hair use cosmetic which contains at least 0.001 weight% of
a resorcinol derivative which is represented by:

General Formula (1)



(In the formula, R represents a straight chain or branched
alkyl group of a carbon number 2~12.)

3. Detailed Explanation of the Invention

(Industrial Field of Application)

This invention pertains to a hair cosmetic and especially pertains to a hair cosmetic which realized dandruff control utilization effects which contains a compound having desirable antimicrobial action.

(Prior Art)

The greatest origin of the production of dandruff is realized to be the propagation of microorganisms and as the degradation origin are the so called irregular [illegible] of the keratin cells and skin secretion promotion. The microorganisms include ones such as *Pityrosporum ovale* (*Pityrosporum ovale*) (Hifu to Biyo, 9(2),1371(1977)).

Thus, cadmium sulfate, io[do] compounds such as zinc pyridinium-1-thiol-N-oxide (Zpt), cadmium sulfate, io[dine], trichlorozan[?], halocarbons, alantoin, mesytol[?] [illegible; probably: groups] and undecylic acids, and, especially, resorcin, isopropylmethyl phenol and salicylic acid are known as prior control agents for dandruff, and these are utilized for the control of dandruff by being contained in shampoos.

(Problems to be Solved by the Invention)

The antimicrobial action for control agents for dandruff in the aforementioned prior art is weak for ones such as *Pityrosporum ovale* which are microorganisms which increase the production of dandruff when propagating and [these] cannot be stated as being substances which have a high dandruff control effect; further, the previous do not obtain great utilization for dandruff control agents and the stability and safety of one such as cosmetic

products which contain these for that [control]

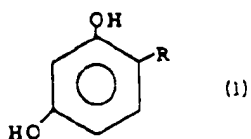
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are problems and the development of a hair cosmetic which does not have problems for the point of safety with a high dandruff control effect is desired.

(Means for Solving the Problems)

These inventors realized this invention by the results of various research for hair cosmetics with high dandruff control effects of characteristic resorcinol derivatives and hair cosmetics containing this attained a dandruff control effect through a strong antimicrobial action for microorganisms having propagation which produces dandruff and for *Pityrosporum ovale*, in particular.

Thus, this pertains to a hair use cosmetic formed as containing at least 0.001 weight% of a resorcinol derivative of this invention and is represented by General Formula (1):



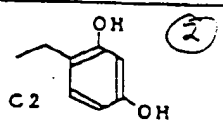
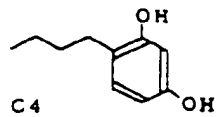
(In the formula, R represents a straight chain or branched alkyl group with a carbon number of 2~12.)

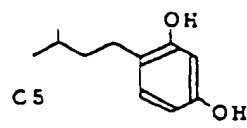
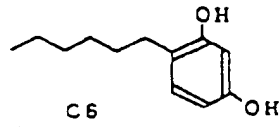
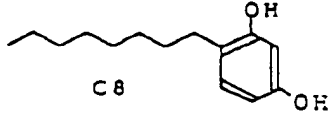
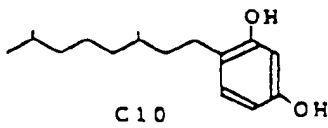
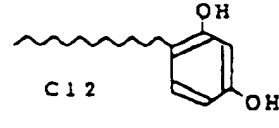
R for the resorcinol derivative which is represented in the aforementioned General Formula (1) is a straight chain or branched alkyl group of carbon number 2~12 and the straight chain alkyl groups can include ones such as ethyl groups, propyl groups, butyl groups, pentyl groups, hexyl groups, heptyl groups, octyl groups,

nonyl groups, decyl groups, undecyl groups and dodecyl groups. also, 1 hydrogen atom can be substituted for a low grade alkyl group such as methyl of a branched alkyl group and concrete examples can include ones such as isopropyl groups, isobutyl groups, isoamyl groups and 2-methylhexyl groups.

Concrete compounds of resorcinol derivatives of this invention are represented through Table 1.

Table 1

番号	化学構造式
① No. 1	
No. 2	

No. 3	
No. 4	
No. 5	
No. 6	
No. 7	

[Key to Table 1]

- 1 number
- 2 chemical structural formula

The resorcinol derivatives represented by the General Formula (1) are well known compounds and can be easily obtained according to ones such as methods which reduce condensation products by zinc amalgam/hydrochloric acid (Lille. J. Bitter, LA. Peiner. V, Tr. Nauch-Tealed, Inst. slantaev 1969, No. 18, 127) and methods which obtain reactions at high temperatures of 200-400° using alumina catalyst for alkyl alcohols related to resorcinol (British Patent Number 1, 581, 428), for example, after combining resorcinol and saturated carboxylic acids in the presence of zinc chloride.

The combined mixture of resorcinol derivatives which are represented by General Formula (1) are at least 0.001 weight% for the hair cosmetic compound total weight, with 0.1-2 weight% being a particularly desirable amount. The dandruff control effect is weak for concentrations lower than 0.001 weight% and there is not the obtainment of the realization of the objective of this invention. The hair cosmetic of this invention

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combines the respective compounds which are commonly used in hair cosmetics for the value of the aforementioned resorcinol derivatives and [these] are not particularly limited.

Concrete formulation of hair cosmetics of this invention can include ones such as shampoos, rinses, hair tonics, hair treatments and hair creams.

The foundations of this hair cosmetic can include 1 type or combinations of 2 types or more of ones such as the respective types of ester groups, rou[?] groups, fat and oil groups, high

grade fatty acid groups, high grade alcohol groups, water soluble polymer groups, powders, surfactants and polyhydric alcohol groups. Further, the added components can include ones such as perfumes, pigments, preservatives, antioxidants, ultraviolet ray absorbants and other various pharmaceutical components such as hyaluronic acid, alantoin, vitamin groups, amino acids and placenta extract. these can be suitably added as 1 type or by mixing 2 types or more.

(Utilization)

A resorcinol derivative of this invention has a strong antimicrobial action for microorganisms which have propagation that produces dandruff and has a dandruff control effect for hair cosmetics which contain this.

(Actual Examples)

this invention is concretely explained in the actual examples, below, but is not limited to these actual examples. Further, the mixed amounts are weight parts.

(1) Antimicrobial Activity Tests of Resorcinol Derivatives

<Test Methods and Results>

(Samples)

Each sample is dissolved in 15% ethanol aqueous solution

(Test Methods)

Protected circle determination is done a cap method

Determination Methods Using a Cap Method

A culture was done in approximately 20 ml of culture medium which contains 0.07% of polyoxyethylene (20) sorbitan monooleate

Afterward, the aforementioned mixed culture was placed in a cap (inner diameter 8 mm) and anaerobically incubated for 2 days at 30°C with 0.3 ml of each sample in the caps. The diameter of the protected circle was measured by removal of the cap at the completion of the incubation. The results are shown in Table 2.

<div style="display: flex; align-items: center;"> <div style="border: 1px solid black; border-radius: 50%; padding: 5px; margin-right: 10px;">②</div> <div style="text-align: center;"> サンプル濃度 サンプル </div> <div style="border: 1px solid black; border-radius: 50%; padding: 5px; margin-left: 10px;">①</div> </div>		<div style="display: flex; align-items: center;"> <div style="border: 1px solid black; border-radius: 50%; padding: 5px; margin-right: 10px;">③</div> プチロスポラム オパール </div>				
		0.05%	0.07%	0.1%	0.5%	1.0%
<div style="display: flex; align-items: center;"> <div style="border: 1px solid black; border-radius: 50%; padding: 5px; margin-right: 10px;">④</div> <div style="text-align: center;"> 本品 説明書 ④ </div> </div>	レゾルシン No.3	8.0mm	9.5	10.8	21.5	22.1
	レゾルシン No.2	8.0	10.0	11.5	23.5	27.5
<div style="display: flex; align-items: center;"> <div style="border: 1px solid black; border-radius: 50%; padding: 5px; margin-right: 10px;">⑤</div> <div style="text-align: center;"> 本品 ⑤ </div> </div>	レゾルシン	0	0	0	0	8.0
	イソプロピルアルコール	0	0	0	0	8.0
	サリチル酸	0	0	0	0	8.0
<div style="display: flex; align-items: center;"> <div style="border: 1px solid black; border-radius: 50%; padding: 5px; margin-right: 10px;">⑥</div> <div style="text-align: center;"> 阻害 (15%エタノール) </div> </div>		0				

- 1 sample concentration
2 sample
3 *Pityrosporum ovale*
4 resorcinol derivative No. 3
resorcinol derivative No. 2
5 resorcin
isopropylmethyl phenol
salicylic acid
6 product of this invention
7 prior [art] product
8 solvent (15% ethanol)

The resorcinol derivatives of this invention have strong antimicrobial action (the protected circle is large) for microorganisms of *Pityrosporum ovale* which produces dandruff when propagating such as is clarified by Table 2. Hair cosmetics which contain these obtained a dandruff control effect.

(2) Acute Toxicity Test

Resorcinol derivatives or hydroquinone as the reference compound was dissolved in physiological saline and these were [respectively] dosed orally (p.o.), into the peritoneum (i.p.), and subcutaneously (s.c.) to ddy-type male mice of 10 per 1 group and then observed for the [ones which are] alive or dead until 24 hours after dosing. The LD₅₀ was calculated by the Litchfield Wilcoxon (Litchfield.Wilcoxon) method based on that results and the results are shown in Table 3

化合物 No. ①	LD ₅₀ (mg/kg)		
	i.p.	s.c.	p.o.
No. 4	334.8	>500	>500
No. 3	268.8	>500	>500
② ハイドロキノン	144.0	338.8	489.0

[Key to Table]

- 1 compound
- 2 hydroquinone

Actual Examples 1, 2 and 3Shampoos

(Treatment)

② ③ ③

原料名 ①	実例			比	
	1	2	3	例1	例2
ヤシ油脂肪酸ジエタノールアミド	2.5	2.5	2.5	2.5	2.5
アルスコープM-35 (東京化学) *1	10.0	10.0	10.0	10.0	10.0
アノンGLM-R (日本油脂) *1	8.0	8.0	8.0	8.0	8.0
ビスタCAP (松本油脂) *3	8.0	8.0	8.0	8.0	8.0
ポリマーJR (ユニオンカーバイド) *4	0.5	0.5	0.5	0.5	0.5
パラオキシ安息香酸メチル	0.2	0.2	0.2	0.2	0.2
エデト酸2ナトリウム ④	0.1	0.1	0.1	0.1	0.1
香 料	0.1	0.1	0.1	0.1	0.1
1,3-ブチレングリコール	8	8	8	8	8
レゾルシノール誘導体 No. 3	0.5	-	0.2	-	-
レゾルシノール誘導体 No. 2	-	0.5	0.2	-	-
イソプロピルメチルフェノール					0.5
精製水	52.1	52.1	52.2	52.6	52.1

[Key to Table]

- 1 raw material
- 2 actual example
- 3 reference example
- 4 coconut oil [illegible; probably: fatty acid] diethanol amide
- Arusuko-bu M-35 (Toho Kagaku) *1
- Anon GLM-R (Nihon Yushi) *2
- Bisuta CAP (Matsumoto Yushi) *3
- Porima-JR (Yunionka-baido) [Polymer JR (Union Carbide) *4
- methyl peroxybenzoate
- sodium edetoate 2
- perfume
- 1,3-butylene glycol
- resorcinol derivative No. 3
- resorcinol derivative No. 2
- isopropylmethyl phenol
- pure water

- *1) 30% aqueous solution of sodium polyoxyethylene lauryl ether
(3E.O.) sulfate
- *2) 2-alkyl-N-carboxymethyl-N-hydroxyethyl imidasolinium[?]
betaine
- *3) 30% aqueous solution of coconut oil fatty acid
amidopropyl dimethylamino acetate betaine
- *4) hydroxyethyl cellulose-hydroxypropyl trimethyl ammonium
chloride ether

(Manufacturing Method)

All are mixed until uniform at room temperature and the product is put into vessels.

<Anti-dandruff Tests>

(Samples)

Shampoos of Actual Examples 1, 2, 3
and Reference Examples 1, 2

(Test Methods)

A group of 6 females with frequent dandruff were taken as the reference group with 3 individuals per 1 group and actual examples were tested on 30 individuals divided into 5 groups. The test period was 1 month with [the hair] being washed once [1] each day and the Reference Example 1 was used as the control for the initial 2 weeks, then the Reference Examples 1 and 2 were used for the reference comparison with the Actual Examples 1, 2 and 3 for the final 2 weeks.

The variations of dandruff amount were done as comparisons of

the dandruff approximate dry weight amount when washing the final 2 times within the control utilization period and the dandruff average dry weight amount when washing 2 times after 2 weeks of washing with a reference product or a product of this invention.

The removed dandruff after washing on the zaru[?] which are covered with impurities and drying the impurities, the hair and dandruff which are removed and filtered and removed are recovered; after removing the hair, the amount of accumulated dandruff which has been dried under reduced pressure is measured. Further, the reduction rate is determined by the following formula.

$$\text{reduction rate} = ((\text{amount of control [illegible] dandruff} - \text{amount of dandruff of [illegible]})) / \text{amount of dandruff after control utilization} \times 100\%$$

(Results)

(i) Cases of Actual Example 1 of This Invention

Table 3

① 被験者	フケ重量 (mg) ②		減少率 (%) ⑤
	コントロール	本発明実施例 1	
1	15.2 ③	5.6 ④	63.2
2	29.5	14.2	51.9
3	25.4	12.9	51.3
4	20.1	18.2	52.3
5	40.6	17.5	56.9
6	73.8	34.2	53.7

第3表から明らかなように、6例共に顕著なフケ抑制効果が認められる。

[Key to Table 3]

- 1 given sample
- 2 dandruff weight (mg)
- 3 control
- 4 Actual Example 1 of This Invention
- 5 reduction rate (%)

A remarkable dandruff control effect can be stated for all 6 examples such as is clarified from Table 3.

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(ii) Cases of Actual Example 2 of This Invention

Table 4

① 被験者	フケ重量 (mg) ②		⑤ 減少率 (%)
	コントロール	本発明実施例 2	
1	20.7	10.5	49.3
2	29.8	14.1	52.7
3	32.5	15.8	51.4
4	50.7	25.0	50.7
5	45.6	22.8	50.0
6	65.1	29.1	55.3

[Key to Table 4]

- 1 given sample
- 2 dandruff weight (mg)
- 3 control
- 4 Actual Example 2 of This Invention
- 5 reduction rate (%)

A remarkable dandruff control effect can be stated for all 6 examples such as clarified from Table 4.

(iii) Cases of Actual Example 3 of This Invention

Table 5

(1) 試験者	(2) フケ重量 (mg)		減少率 (%) (5)
	コントロール	本発明実施例 3	
1	21.1	10.5	50.2
2	33.7	17.2	49.0
3	28.4	14.7	48.2
4	55.7	28.2	47.6
5	70.3	33.5	49.5
6	63.4	31.6	50.2

[Key to Table 5]

- 1 given sample
- 2 dandruff weight (mg)
- 3 control
- 4 Actual Example 3 of This Invention
- 5 reduction rate (%)

A remarkable dandruff control effect can be stated for all 6 examples such as clarified from Table 5.

(iv) Cases of Reference Example 1 for Reference Comparison

Table 6

① 試料番号	② フケ重量 (mg)		減少率 (%) ⑤
	コントロール	比較例 1	
1	35.7	34.3	3.9
2	24.2	25.9	-7.0
3	31.6	33.7	-5.7
4	47.2	45.8	3.0
5	63.5	62.1	2.2
6	59.6	58.4	2.0

[Key to Table 6]

- 1 given sample
- 2 dandruff weight (mg)
- 3 control
- 4 Reference Example 1
- 5 reduction rate (%)

No reduction rate can really be stated for the dandruff control effect for any of the 6 examples by the reference products which do not contain resorcinol derivatives such as clarified from Table 6.

(v) Cases of Reference Example 2 for Reference Comparison

Table 7

① 試験者	フケ重量 (mg) ②		⑤ 減少率 (%)
	コントロール	比較例 2	
1	29.3 ③	27.9 ④	4.8
2	38.6 ③	35.2 ④	8.8
3	33.7	29.1	13.6
4	64.2	58.7	7.0
5	43.5	40.0	8.0
6	52.4	48.6	7.3

[Key to Table 7]

- 1 given sample
- 2 dandruff weight (mg)
- 3 control
- 4 Reference Example 2
- 5 reduction rate (%)

The dandruff control effect is low for all 6 examples by the reference product containing isopropylmethyl chi[?] phenol which is used previously such as clarified from Table 7.

Actual Example 4 Hair Tonic

(Treatment)

Table 8

原料名 ①	実施例 4 ②	比較例 3 ③
エタノール ④	65	65
1,3-ブチレングリコール	2	2
メントール	0.2	0.2
ポリオキシエチレン脂肪酸エステル (50.E.O.)	1	1
香料 ⑤	0.05	0.05
レゾルシノール誘導体 No. 2	0.1	-
精製水	31.65	31.75

[Key to Table 8]

- 1 raw material
- 2 Actual Example 4
- 3 Reference Example 3
- 4 ethanol
- 1,3-butylene glycol
- menthol
- polyoxyethylene [illegible] castor oil (50.E.O.)

(Manufacturing Method)

All are stirred until becoming uniform at room temperature then the product is put into vessels.

<Anti-Dandruff Tests>

(samples)

The hair tonics of Actual Example 4 and Reference Example 3

(Test Methods)

The actual testing was by 6 individuals which were women with frequent dandruff divided, respectively, to 3 women per group for reference.

The test period was 1 month with washing being performed with the Reference Example 1 on each [1] day and the Reference Example 3 was utilized after washing for the initial 2 weeks, and the hair tonic of Actual Example 4 of this invention was utilized after washing for 2 weeks after that.

The removal method for the dandruff was through the previous for methods which determine the reduction rate of the dandruff.

(Results)

Table 9

① 被験者	② フケ重量 (mg)		⑤ 減少率 (%)
	コントロール	本発明実施例 4	
1	24.1	10.4	56.8
2	31.7	12.6	60.3
3	27.6	11.4	58.7
4	49.3	18.5	62.5
5	66.2	25.6	61.3
6	58.5	23.8	59.3

[Key to Table 9]

- 1 given sample
- 2 dandruff weight (mg)
- 3 control
- 4 Actual Example 4 of this invention
- 5 reduction rate.

A remarkable dandruff control effect can be stated for all 6 examples such as clarified from Table 9.

(Effects of the Invention)

A hair cosmetic which contains at least 0.001% resorcinol derivative can be stated as having a remarkable dandruff control effect and the point of safety is realized.

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Continued from Page 1

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/89

Procedural Revision

September 30, 1991

Director General of the Patent Office

Mister

1. Represented Patent

Hei 2-291880

2. Title of the Invention

Hair Cosmetic

3. Person Revising

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[stamp]

5. Date of Revision Order Voluntary Revision

6. Number of Inventions Added by Revision None

7. Subject of Revision

Section of "Detailed Explanation of the Invention".

8. Revision Content

(1) Specifications Page Number 9

Entry through the following of Table 2

Table 2 Protected Circle (mm)

① サンプル濃度		ブチロスポラム オバール ②				
サンプル		0.01%	0.05%	0.1%	0.5%	1.0%
本	レゾルシン No. 1	8.0mm	8.0	8.4	17.8	18.7
異	レゾルシン No. 2	8.0	10.0	11.5	23.5	27.5
明	レゾルシン No. 3	8.0	9.5	10.8	21.5	22.1
品	レゾルシン No. 5	8.0	9.0	10.1	18.5	18.3
③	レゾルシン No. 7	8.0	8.0	8.0	13.2	13.5
従	レゾルシン	0	0	0	0	8.0
④	イソプロピルフェノール	0	0	0	0	8.0
品	サリチル酸 ⑥	0	0	0	0	8.0
溶媒 (15%エタノール)		0				

⑦

[Key to Table 2]

- 1 sample concentrations
- sample
- 2 *Pityrosporum ovale*
- 3 product of this invention
- 4 prior [art] product
- 5 resorcinol derivative [No. 1, 2, etc.]
- 6 resorcin
- isopropylmethyl phenol

salicylic acid

7 solvent (15% ethanol)

(2) attached from below row number 5~bottom row on the same page number 9

Revision through the following of the entry of "Table 2...being one which is."

"The protected circle is smaller for No. 1 (carbon number 2) and No. 7 (carbon number 12) than for No. 2 (carbon number 4), No. 3 (carbon number 5) and No. 5 (carbon number 8) for the carbon number 2~ for R in the formula of the resorcinol derivatives of this invention but are larger than the prior [art] products and the antimicrobial effect for the microorganism *Pityrosporum ovale* which propagates generating dandruff is strong (the protected circle is large) for carbon numbers of 2~12 of R in the formula of the resorcinol derivatives of this invention such as is clarified from Table 2, and hair cosmetics containing these are understood to realize a dandruff control effect.